

APPLICATION UNDER UNITED STATES PATENT LAWS

Atty. Dkt. No. 46104

Invention: CONTROLLED RELEASE AGRICULTURAL PRODUCTS AND PROCESSES FOR
MAKING SAME

Inventor (s): Taylor Pursell
Arthur R. Shirley, Jr.
Keith D. Cochran
Timothy G. Holt
Gregory S. Pedeen

Manelli Denison & Selter, PLLC
2000 M Street, NW
Seventh Floor
Washington, DC 20036-3307
Attorneys
Telephone: (202) 261-1000

This is a:

- Provisional Application
- Regular Utility Application
- Continuing Application
- PCT National Phase Application
- Design Application
- Reissue Application
- Plant Application
- Substitute Specification
 - Sub. Spec Filed _____
 - in App. No. /
- Marked up Specification re
Sub. Spec. filed _____
In App. No. /

SPECIFICATION

CONTROLLED RELEASE AGRICULTURAL PRODUCTS AND
PROCESSES FOR MAKING SAME

BACKGROUND OF THE INVENTION

5 This invention relates to controlled release agricultural products and processes for making such products. More particularly, the present invention is directed to particulate absorbents in particulate form and holding compositions in particulate form that provide for controlled release of

10 agriculturally beneficial materials such as fertilizers, insecticides, herbicides and fungicides. The particulate absorbents contain capillaries/voids between 10-200 microns in cross-sectional diameter and are impregnated in an amount of 40-95 % of the capillaries/voids volume with the agriculturally beneficial material. The process of the

15 present invention forms the controlled release agricultural particulate absorbents by blending the absorbent with the agriculturally beneficial material(s) for a prescribed time.

The blended absorbent is fed into a granulator and after

20 screening, the product is dried. The process results in an easily handled, free flowing, controlled release agricultural absorbent based product.

There are many slow and extended release fertilizers with their nutrient release based on time and event related coating

25 failures or coating permeability, and/or low solubility, and/or microbial activity in the soil, and/or a ratio of

surface area to nutrient weight of the particle. Of these, the major commercial products are sulfur coated urea, polymer coated ureas, and urea - formaldehyde products such as methylene ureas. The production costs of these materials

5 vary, but all of these commercially available products have been judged by the consumers' spending patterns as too expensive for extensive use in agriculture. This especially occurs in the case of major crops such as wheat and corn which are grown on moist, dry, and irrigated soils under varying weather conditions. In addition, these high priced extended release products are in general not tailored for the short growing periods of wheat and corn because they do not release their nutrients completely within the growing period of these crops. The existing products are tailored for severe water regime nutrient applications, such as rice, sugar cane, and pineapples and high-end truck farm crops, such as strawberries, and cranberries, or shrubs, ornamentals, and flowers. When used for grasses, existing products are limited because of their high cost for use on lawns, gardens, parks,

10 golf courses and commercial, governmental, and educational grounds. The existing products are not extensively used for pasture lands because of the added processing cost of the fertilizer. Furthermore, when existing controlled release products are used for lawn care applications, many purchasers-

15 users primarily desire burn protection caused by overdosing the lawn while applying the fertilizer. They do not

necessarily desire long term release properties that the fertilizer may provide.

Thus, a low cost alternative fertilizer with a much shorter controlled release period would be superior to the
5 higher cost, longer controlled/extended release fertilizers.

BRIEF SUMMARY OF THE INVENTION

The present invention includes numerous embodiments of a controlled release agricultural absorbent based product. The absorbent based product includes particles of an absorbent material containing capillaries/voids between 10-200 microns in cross-sectional diameter which is impregnated in an amount of 40-95 % of the capillaries/voids volume with an agriculturally beneficial material selected from the group consisting of fertilizers, insecticides, herbicides and fungicides. The absorbent material includes for example, expanded perlite, shredded newspaper, saw dusts, cotton lint, ground corn cobs, corn cob flower, Metrecz absorbent and
20 diatomaceous earth.

The fertilizer includes nitrogen compounds, phosphorous compounds and potassium compounds. The nitrogen compounds include urea, ammonia, ammonium nitrate, ammonium sulfate, calcium nitrate, diammonium phosphate, monoammonium phosphate,
25 potassium nitrate and sodium nitrate. The phosphorous compounds include diammonium phosphate, monoammonium

phosphate, monopotassium phosphate, dipotassium phosphate, tetrapotassium pyrophosphate, and potassium metaphosphate.

The potassium compound includes potassium chloride, potassium nitrate, potassium sulfate, monopotassium phosphate,

5 dipotassium phosphate, tetrapotassium pyrophosphate, and potassium metaphosphate.

The agriculturally beneficial material also includes micronutrients, secondary nutrients, growth regulators, nitrification regulators, as well as the aforementioned insecticides, herbicides and fungicides.

The particles of absorbent may be agglomerated into granules of a predetermined size.

An important embodiment of the invention is the impregnation of the particle absorbent with a mixture of an interspatial blocker and the agriculturally beneficial material. The interspatial blocker includes plant starches, protein gels, glues, gumming compositions, crystallizing compounds, gelling clays, and synthetic gel forming compounds.

The presence of the interspatial blocker acts to regulate the
20 release of the agriculturally beneficial material.

Another embodiment of the present invention includes A controlled release, particulate, agricultural product that includes a mixture of a control release holding substance, such as plant starches, protein gels, glues, gumming
25 compositions, crystallizing compounds, gelling clays and synthetic gel forming compounds, and an agriculturally

beneficial material including fertilizers, insecticides, herbicides and fungicides.

The present invention also includes embodiments of processes for making the controlled release agricultural absorbent based product. The process includes, for example, the steps of 1) introducing water to particles of absorbent material to result in absorption of water within the absorbent material, 2) heating the absorbent particles and water to transform the water within the absorbent particles to steam, 3) introducing the heated absorbent particles to an agriculturally beneficial material in aqueous solution to essentially saturate the absorbent particles with the agriculturally beneficial material, 4) granulating the combination of agriculturally beneficial material and saturated absorbent particles to solidify and harden the mixture, resulting in the agglomeration of absorbent particles into granules, and 5) drying the granules.

The present controlled release agricultural absorbent based product and holding material based product provide for fine control of the release over both short and long periods of time, for a variety of agriculturally beneficial materials.

BRIEF DESCRIPTION OF THE DRAWINGS

Further objects and advantages of the present invention will be better understood by carefully reading the following

detailed description of the presently preferred exemplary embodiments of this invention in conjunction with the accompanying drawings, of which:

5 FIGURE 1 is a flow chart showing one embodiment of the process of the present invention wherein a controlled release agricultural absorbent based product is produced containing fertilizer and a gel forming interspatial blocker.

10 FIGURE 2 is a photomicrograph showing expanded perlite wherein the particles appear to be covered with a thin shell.

15 FIGURE 3 is a photomicrograph showing exfoliated perlite wherein the internal capillaries and voids are exposed.

FIGURE 4 is a photomicrograph showing the exfoliated perlite of FIGURE 3 at higher magnification to observe the greater exposure of internal capillaries and voids.

20 FIGURE 5 is a photomicrograph showing the expanded perlite of FIGURE 2 at the higher magnification as in FIGURE 4 in order to compare the relatively closed surface compared to the exfoliated perlite of FIGURE 4.

FIGURE 6 is a schematic showing the plant growth test plots of Example 16, demonstrating the utility and effectiveness of the products of the present invention.

5 DETAILED DESCRIPTION OF THE INVENTION

One embodiment of the present invention is the newly developed controlled release fertilizer which extends the release of plant nutrient from absorbent particles over a period of time by trapping the plant nutrients in the small capillaries and voids of an absorbing material. Techniques utilize innovative means to provide deep penetration and extensive absorption of an agriculturally beneficial material into the absorbent material. Where this absorbed material contains plant nutrient, the result is a fertilizer with controlled nutrient release characteristics. In most cases, we have been able to further enhance the retention of the nutrient within the absorbent through use of an interspatial blocker such as a gelling compound, which helps further trap the nutrient within the small capillaries and voids of the absorbent material. We have tried many absorbents and methods of absorption, along with several gel forming materials, with varying levels of controlled nutrient release. We have been the most successful where the absorbents are extremely absorbent which results in a relatively dense concentration of nutrient. For testing and development purposes, urea was

selected as representative of nutrient/fertilizer agriculturally beneficial materials. It was the nutrient most tested. Best results have been achieved when using perlite as the absorber, although milled newspaper and fine pine sawdust 5 have been very good as absorbents. Utilizing cornstarch as the interspatial blocker (a gelling substance) has improved controlled nutrient release.

Pollution is an ever increasing problem with respect to both air pollution and water pollution. Water pollution 10 occurs when readily soluble fertilizer is solubilized and washed into streams during rains or is solubilized and is leached into the ground water before its intended target vegetation is able to capture. Failure to capture the fertilizer occurs because the target vegetation is not in need of it when it becomes soluble or because the leaching rate is 15 too rapid. Some fertilizers, in particular urea, are lost to the atmosphere through volatilization where urea decomposes to ammonia, carbon dioxide, biuret, and other volatile compounds. Therefore, since the vast majority of fertilizer used has no 20 controlled release properties because they are not available at a low cost, pollution problems are being caused by inefficient use of soluble and volatile fertilizers, which must be applied in excess amounts over the crop's need.

Those who are familiar with the production, storage, 25 transportation, and application of fertilizers know that the nutrient concentration and the physical properties of a

fertilizer are extremely important in its acceptance and use by the agricultural community.

Our invention addresses the problems of production, storage, shipping, and application costs, as well as the need 5 for moderation in the length of nutrient availability from slow and controlled release fertilizers. It provides a process that produces a high analysis granular material, for example 40 to 45% by weight nitrogen when using perlite and urea, with or without corn starch, at an extremely low production cost for a controlled release fertilizer.

Concurrently, the invention provides a product with physical properties equal to and for the most part more desirable than commercially available urea.

The nutrient strength of commercial urea is commonly recognized as 46-0-0, which is 46% nitrogen. The most common slow release nitrogen, sulfur coated urea, varies from 32% nitrogen to 38% nitrogen depending on its size and the thickness of the coating it is given to obtain the desired release rate. Therefore, substantially more weight (typically 20 28% more) of sulfur coated urea is required to provide the same amount of nutrient. When this property of a fertilizer is coupled with the physical property commonly called bulk density, which is the amount of weight which occupies a unit of volume, e.g. lbs/ft³, then we have the full impact on the 25 cost of storage and distribution of the fertilizer. In the

case of urea and sulfur coated urea, the bulk density is about the same at 45 to 46 lb/ft³.

To achieve a fertilizer which will be accepted by the agricultural community as a replacement for urea and sulfur coated urea, we have developed products which approach the bulk density and exceeds the crushing hardness of urea.

Handling characteristics are much better than for sulfur coated urea. Handling and storage do not affect the controlled release properties of our product, but they can,

for example, crack the coating of sulfur coated urea. Our product varies in nitrogen strength from 40.0% nitrogen to 45.0% nitrogen, with a more preferred range being 43.0% nitrogen to 44.0% nitrogen. At the same time we had been able to perfect the nutrient absorption and granule forming aspects of the product such that bulk densities have been achieved from 25 lb/ft³ to 43 lb/ft³, with a more preferred range being 35 lb/ft³ to 46 lb/ft³ and the most preferred range being 38 lb/ft³ to 46 lb/ft³. The concentrations of nitrogen using urea and perlite and those bulk densities of final product have

been achieved in a laboratory and a pilot plant while maintaining the controlled release properties of the fertilizer. In using larger equipment, as in a full scale plant, with the techniques taught herein, the bulk density is 46 lb/ft³, the same as that of urea and sulfur coated urea while maintaining 44% nitrogen content of the fertilizer and the controlled release aspects of our product.

Several innovative methods were developed to increase the density of the resulting controlled release fertilizer. Such methods provide a superior, concentrated product, having improved handling characteristics and controlled release properties. The product should have a bulk density approaching that of urea to provide economics of storage, transportation and distribution near or equal to those of urea.

In one embodiment of the present invention, our dense, concentrated product is accomplished by the following important features: 1) already expanded perlite is further steam exfoliated beyond its normal popped form to allow better penetration and filling of its interspatial regions by the urea/corn starch mixture; 2) urea/corn starch melts are maintained around 95 to 98% concentration to minimize voids formed from evaporation during the processing; and 3) the small perlite particles containing urea/corn starch are granulated together to form dense, spherical particles.

In general, the process involves taking a proper absorbent material and a fertilizer melt or solution and absorbing the fertilizer melt or solution (which is in a dense saturation state) into the absorbent material and then solidifying the fertilizer within the voids of the absorbent such that it is difficult for the fertilizer to be released by the absorbent when in contact with water or humid conditions. This is done by utilizing a very absorbent material with small

capillaries and/or voids and accomplishing the absorbance by keeping the fertilizer and the absorbent above the fertilizer's initial crystallization temperature and at viscosities where capillary action easily occurs while

5 absorption is occurring. For improvements in controlled release characteristics, an interspatial blocker, such as starches and/or other gelling compounds are homogenized into the fertilizer melt or solution before the absorption step of the process. When solidified, these gelling compounds tend to

10 help trap the soluble fertilizer nutrients within the capillaries and/or internal voids of the absorbent. Following absorption and prior to crystallization of the fertilizer melt or solution within the absorbent, the liquid filled absorbent is mixed with recycled material, previously crystallized, to

15 solidify and granulate the liquid filled absorbent with the recycled material through cooling and/or drying, at least partially, imparted by these recycled materials within a pugmill, drum, rotating pan, fluid-bed, or similar standard granulation equipment or combination of standard granulation

20 equipment. Before being stored as product, the granulated solids are milled, screened, further cooled and dried, but not necessarily in that order, by any of the obvious ways before sending the product to storage. The material also is easily prepared using the solids forming techniques which do not use

25 recycle of solid particles for cooling, such as slating, prilling, rotoforming, low pressure extrusion, molding, and

forming of bulk slabs or molded shapes. As needed, any of these methods can involve milling of the obtained solids with screening and further cooling and drying as needed with fines recycled to the starting melt or solution filled absorbent for 5 inclusion in the solidification process. The cooling and drying can be accomplished by the use of most all standard methods presently known in the art of granulation including, but not limited to direct gas contact, vacuum enhanced evaporation, and indirect heat exchange.

Although the development can extend to many fertilizer nutrients, we have centered our development work to date on providing controlled release urea. Other nutrient fertilizers which can be used to provide controlled release fertilizer include, but are not limited to the following; ammonia, ammonium nitrate, ammonium sulfate, calcium nitrate, diammonium phosphate, monoammonium phosphate, potassium chloride, potassium nitrate, potassium sulfate, potassium phosphates, such as monopotassium phosphate, dipotassium phosphate, tetrapotassium pyrophosphate, and potassium metaphosphate, and sodium nitrate and combinations of these materials. The urea melt is maintained between 40% and +99.9% by weight urea; however, a preferred range of the melt would be between 65% and +99.9% and a most preferred range between 75% and +99.9% by weight urea. To provide other controlled 20 release fertilizer, one or more other nutrient materials other than urea can be absorbed as long as the nutrients are in the 25

fluid phase by being pure melt or by being solubilized in water or in the melt of another nutrient or combination of nutrients and/or water. For example, a full NPK fertilizer can be made by using urea, monoammonium phosphate, diammonium phosphates, and potassium chloride in various proportions and concentrations, and then blending the product with a filler to provide, for example, 29-3-4, 16-4-8, 10-10-10, 15-5-10, 15-0-15, 22-3-14, 20-28-5, and 12-6-6 control release fertilizers.

Further, the nutrients can be in the fluid phase by being in a volatile substance such as e.g. ethanol or methanol as the solvent, which can be evaporated out as the material is solidified and dried. In the above manner, it is possible to prepare controlled release fertilizers containing various mixtures of nitrogen, phosphorus, and potassium as well as incorporation of various secondary nutrients (e.g. sulfur, calcium, and magnesium) and micronutrients (e.g. boron, copper, iron, manganese, molybdenum, zinc) if not all of the secondary and micronutrients, and secondary and micronutrients as well as growth regulators such as, but not limited to,

20 potassium azide, 2 amino-4-chloro-6-methyl pyrimidine, N-2, 5-dicorphenyl succinamide, 4-amino-1, 2,4-triazole hydrochloride and nitrification regulators such as, but not limited to, 2-chloro-6-(trichloromethyl)pyridine, sulfathiazole, dicyandiamide, thiourea, and guanylthiourea.

25 The controlled release absorbent particles are small and must be granulated for most commercial application. It is

possible to granulate the filled absorbent particles either in their liquid filled or solidified condition with other non-absorbed materials to give controlled release properties to only that portion of the material contained in the absorbent.

5 See Figure 1 for a flow diagram of one embodiment of the processes of our invention. The blending of cornstarch and urea, if needed, is done through the use of high shear agitation provided by a homogenizer. The cornstarch addition in tests carried out in the laboratory and pilot plant has worked well. Cornstarch addition can range from 0.01 to 20% by weight cornstarch, with the preferred range being from 0.2 to 10% by weight cornstarch and the most preferred range being from 0.5 to 4% by weight cornstarch. The homogenized mixture is mixed (poured not sprayed) gently with the exfoliated and/or expanded perlite at near the full absorbing capability of the perlite, which is approximately 4 to 7.5% for our exfoliated perlite, by weight of the final product; thus the absorbed urea and cornstarch makes up approximately 95% of the weight of the final product. Prior to the mixing, the

10 exfoliated and/or expanded perlite is preheated significantly above the melt temperature to prevent premature freezing of the melt before full penetration into the perlite. Preheating expanded perlite to temperatures as high as 330°F has been successful. Preheating of the perlite is desirable but we

15 have made a good product just by keeping the mixture of perlite and melt above the melting point of the urea melt or

solution while absorption is occurring. Although full absorption of the urea and fertilizer into the exfoliated perlite or other absorbent is the preferred manner for most products, a reduction in the absorbance will provide a
5 material with less release extension and can be desirable for some fertilizers.

10
15
20
25

We have been successful by 1) submerging preheated exfoliated and/or expanded perlite in an excessive amount of homogeneous urea/corn starch melt or urea melt, and then extracting the fully absorbed particle from the homogeneous melt for granulation; 2) pouring the homogeneous urea/cornstarch melt or urea melt into the preheated exfoliated and/or expanded perlite with gentle mixing until the absorbing capacity of the perlite is obtained before granulation; or 3) in mixing simultaneously metered amounts of exfoliated and/or expanded perlite and urea/cornstarch melt or urea melt and blending them together with gentle mixing while maintaining the melt and perlite above the solidification point of the melt before granulation. Then for all three
20 methods of blending, the resulting blended material is added directly to a pan/drum granulator or pugmill, with recycle and allowed to agglomerate and solidify into granules or is premixed with or without recycle before adding it to the pan/drum. The resulting granules are screened and the
25 oversize is milled and recycled to the screen. The undersize is recycled back to the granulator where it is agglomerated

with the incoming mixed material. In another option, we have been successful in returning the oversize directly to the perlite urea/corn starch mixing step, Vessel 2. Steam can be used to enhance granulation, but our laboratory tests have not 5 shown this is required. The product granules are quickly and easily dried in a pilot plant or laboratory fluid-bed operating with approximately 190°F entering air. The dried granules are then cooled and conditioned against caking, if necessary, before going to storage.

10 All of the exfoliated and/or expanded perlites we have used have worked well. The inside microstructure of an exfoliated and/or expanded perlite particle is comparable to a honeycomb type arrangement; the individual cells indicate diameters of 10 to 200 micron, with a preferred range being 25 to 150 microns, and the most preferred range being 40 to 100 microns. As such, the exfoliated and/or expanded perlite used can have a loose weight density of from 2 to 20 lb/ft³ with a preferred range of 2 to 10 lb/ft³ and a most preferred range of 2 to 6 lb/ft³.

20 One skilled in the art readily will see that the agglomeration and otherwise granule forming, drying, milling, and screening portions of the process are similar to that of a pan/drum agglomeration type granulation process and that of a fluid-bed or prilling granulation process and as such the 25 innovative portion of our process can be easily incorporated into existing and idle fertilizer granulation plants. See the

dashed line enclosure of Figure 1 for the existing plant equipment. Tests have shown that drying can be performed at lower temperatures and without the use of fluid-beds, e.g. within a standard rotary drying drum. The milling step which 5 is obviously forbidden in coated products appears to have no significant adverse effect on the controlled release characteristics of this new invention when milling uses a knife bladed hammer mill similar to those used in large urea granulation plants.

For the most economical process, it is preferred to have the urea as a melt of concentration around 78 to 85%. The urea can be taken directly from the urea synthesis plant and does not need to pass through an evaporator, concentrator per the normal route toward granulation or prilling, hence biuret formation which occurs in the normal granulation urea process of melt concentration and then granulation at high temperatures is avoided. Further, the added costs for production of a controlled release urea fertilizer over that of just urea granules is only the cost of the perlite and, if 10 used, the cornstarch or other gelling additive, and the cost of mixing them with the urea. However for more dense products with enhanced controlled nutrient release characteristics, and the use of less absorbent, we teach the use of higher concentration melts up to 99.9% melt.

25 The products made by our invention continue to retain excellent handling characteristics with regard to hardness and

abrasion resistance and can be made in all size ranges desired by the lawn and garden users as well as the agricultural users. In some cases, by using 78% to 85% by weight urea melt, we can achieve better penetration of the cornstarch
5 within the capillaries and voids of the absorbent material than with +99.9% melt. This increased penetration is apparently due to several reasons; among them lower viscosity of the homogeneous mixture, almost no foaming of the mixture with cornstarch during processing, and reduced pre-gelling of
10 the cornstarch prior to entrance into the exfoliated and/or expanded perlite. When the absorption is done without cornstarch or any additive absorbed into the perlite using the same methods as with cornstarch, significant reductions in controlled release characteristics occur.

All absorbents will not work; it now appears that only those with capillaries and voids between 10 and 200 microns in cell diameter can be used. Further it appears that others, which may work from a controlled release standpoint, have much too small an absorbing capacity, greatly diluting the nutrient
20 content of the fertilizer particle and thus increasing the cost.

In our work to date we have made granular-product of a size from 1mm to 4mm; however, we have made granules ranging in size from 0.20mm to 25mm. These larger and smaller
25 granules have control release properties and product of this size can be made with only a change in the process screen

- size. It is preferable to have granules of about 0.20mm when producing a product to be used on golf greens. The 25mm product would be used in rice patties. The most useful range for lawns and most agriculture is 1mm to 4mm granules.
- 5 Material with a size of 6mm to 8mm will be useful for forestry fertilization.

The urea used can contain normal conditioning additives like formaldehyde, previously reacted urea formaldehyde, clays, ligno products, or parting agents. The presently produced product has shown some excellent handling characteristics. Unlike some controlled release products, it has little tendency to float and it can be blended with most other fertilizers or used directly without blending.

We have successfully made a product using urea melt concentrations up to +99.9% urea melt without cornstarch addition, but at a loss of some controlled release characteristics and some good physical properties because of the absence of the corn starch. Further processing at above about 98% urea concentration leads to excessive formation of 20 biuret, a compound which is undesirable to many agricultural users because of its toxic properties with some crops, in particular, citrus crops. This requires preheating and/or keeping the absorbent above the solidification point of the urea melt and preferably about 20 to 30°F above that point.

25 In our work along with the granulation process techniques otherwise mentioned, we have experienced success in making the

controlled release absorbent based product when using a compaction step, by making a homogenous mixture of cornstarch and urea, or using just urea and then mixing the mixture with the perlite or other absorbents, i.e. shredded newspaper,

5 various saw dusts, cotton lint, ground corn cobs, corn cob flower, Metrecz absorbent, diatomaceous earth, and others.

Then we solidify the material by pouring it out on a flat metal sheet to cool. Following this, the product is milled to the desired particle size; however, when employing a

10 compaction step it is typically milled and compacted into the desired particle size. The controlled release characteristics of the product are usually reduced by the compaction step.

15 Many other pure nutrients and combination of nutrients can be made utilizing the process techniques taught by our disclosure.

In further embodiments of this invention, insecticides such as 0,0-diethyl O-(2-isopropyl-6 methyl- 4 pyrimidinyl) phosphorothioate), herbicides such as 2,4-

dichlorophenoxyacetic acid, fungicides such as ferric-di-

20 methyl-dithiocarbamate, growth regulators such as gibberellic acid, and other agricultural chemicals such as methiocarb can be added during the absorption phase of this process to obtain controlled release characteristics to a complete set of a crop's chemical and nutrient needs. Table 1 includes some 25 more of these chemicals, but those that can be added to the

product during the absorption phase are not limited by this list.

- Other plant starches, protein gels and glues, gumming products, crystallizing compounds, gelling clays, and
- 5 synthetic gel forming compounds also work as the gelling and/or inter-spatial blocking compound. These include but are not limited to the following: rice starch, potato starch, wheat starch, tapioca starch, and any starch which contains the D-glucopyranose polymers, amylose and amylopectin;
- 10 modified starch of the former listing (also including corn starch) by acetylation, chlorination, acid hydrolysis, or enzymatic action which yield starch acetates, esters, and ethers; starch phosphate, an ester made from the reaction of a mixture of orthophosphate salts (sodium dihydrogen phosphate and disodium hydrogen phosphate) with any of the listed (also including corn starch) starch/or starches; gelatin as made by hydrolysis of collagen by treating raw materials with acid or alkali; glue as made from any of the following: collagen, casein, blood, and vegetable protein such as that of soybeans;
- 15 gumming products such as cellulosics, rubber latex, gums, terpene resins, mucilages, asphalts, pitches, hydrocarbon resins; crystallizing compounds such as sodium silicate, phosphate cements, calcium-oxide cements, hydraulic cements (mortar, gypsum); gelling clays in the form of very fine
- 20 powders; synthetic gel forming compounds such as polysulfide sealants, polyethylene, isobutylene, polyamides, polyvinyl

acetate, epoxy, phenolformaldehyde, urea formaldehyde, polyvinyl butyral, cyanoacrylates, and silicone cements.

Plant starches work particularly well, especially corn and wheat starches.

- 5 All granules made can be rounded and/or coated, if desired, with hydrophobic materials such as waxes, polymers, or oils to further enhance their controlled release characteristics.

Scanning electron photo micrographs of our expanded

- 10 perlite showed the expanded perlite to be an in-depth formation of small micro sized chambers connected by walls which are about 0.5 micron thick which formed when water evenly dispersed in the unexpanded perlite expanded under high temperature. For the most part, the expansion of the perlite particles, which are sized before expansion by milling the larger mineral rock, result in particles which appear to have outer shells with blow-holes in the shells. This original perlite expansion can be done by any one of several known technologies. We find that though the resulting expanded
- 20 perlite has potential, it does not allow us to produce the dense product we desire. Therefore, we subject the expanded perlite to further treatment in our pilot plant. A small quantity of water is applied to the expanded perlite, our most preferred amount being from 0.5 ml of water/gm of perlite to
- 25 5.0 ml of water/gm of perlite. The treated expanded perlite is then introduced into a heated chamber, most preferably a

steam jacketed double shaft pugmill running at a high rate of speed so as to mechanically fluidize the particles. This heats the wetted expanded perlite up again such that the water in the perlite expands within the perlite but this time in a much more gentle fashion than the original high temperature and pressure popping technique used in the original expansion.

Air temperatures within the vessel can range from 210°F to 500°F with the most desired range being 215°F to 350°F. The result as shown by the electron microscope is increased
rupture and exfoliation of the outer shell as the absorbed
water expands into steam at atmospheric pressure. There appears to be less effect on the vast maze of internal chambers. The retention time that the wetted perlite spends in the expansion chamber (or pugmill) needs only to be about 30 seconds, but extensive exposure of over an hour is not detrimental unless the mechanical action is too violent and abrades the perlite. The perlite with this enhancement to the original expansion is now ready to be filled with our urea/corn starch mixture. This step of controlled exfoliation of the perlite with steam immediately before it is introduced to the absorbing vessel also drives most of the air from the internals of the previously expanded perlite replacing it with steam. Since urea and urea solutions are extremely hydrophilic as are most fertilizers, the steam in the perlite is absorbed by the fertilizer mixture causing a psuedo vacuum within the perlite which further assists complete filling of

the perlite with urea/corn starch solution or melt when the perlite is fully immersed in the molten material. We have achieved the same exfoliated results in the laboratory using a small tank fitted with a condenser, in a pressure cooker and 5 with a microwave oven. In each case, to get a further rupture of the outer skin of the expanded perlite, water had to be applied to the expanded perlite prior to heating. Scanning electron photo micrographs and calculations, based on percentage of components in the final product and bulk density in the final product, indicate that in the final product, the exfoliated perlite is impregnated to between 40 and 95% of its holding capacity and in most cases, impregnation is between 60 and 90% of its holding capacity. In the most preferred cases, impregnation is between 80 and 90% of the capillaries/void volume. Thereby, the impregnated mixture makes up 70 to 95% by weight of the final product. About 60 to 80% of the urea/corn starch mixture is absorbed into the exfoliated perlite. The remaining urea and corn starch acts as a binder holding the individual granules together and that urea is 20 available for quick release to the soil.

Another major contributor to the high bulk density is the fact that we can granulate the material in the same manner as urea is presently granulated. This is accomplished by spraying the mixture consisting of molten urea, corn starch, 25 and the small perlite particles containing absorbed urea/corn starch mixture, and which vary in size from about 100 micron

to 1500 micron in diameter, but more preferably 150 to 1000 microns, onto existing recycle granules in a rotating drum. The existing granules thus grow in size because of the onion skin type build-up from direct solidification of the mixture sprayed on them and because there is some agglomeration of small existing granules in the rotating bed being adhered to large granules by the solidifying mixture which acts as an adhesive. By such a manner the granules are made spherical. They are then sized as they leave the granulator as per a typical urea granulation plant, with the undersize being returned to the granulator and the oversize being milled and returned to the granulator either in total or just the undersize part after rescreening. The resulting product is spherical even though each granule is made up of a multiplicity of perlite particles filled with solidified urea and starch and the unabsorbed urea and starch acting as the adhesive to hold the granule together. Later when the granules are applied to the soil and water begins to leach-out the urea nutrient, the corn starch not only acts as a inter-spatial blocker thus retarding the leaching of the urea it helps hold the perlite particles together which also enhances controlled release of the nutrient by, in effect, maintaining a larger center of high nutrient content, rather than allowing the dispersion of the small perlite particles in the soil.

Also, in effect, maintaining a large urea granule which

obviously goes into solution slower than the same granule ground to a powder and dispersed in the soil.

Our granules are extremely hard when made at high density even without the customary inclusion of 0.3% to 0.5% urea formaldehyde in urea granules to harden them up and prevent caking. The exfoliated perlite super-structure apparently gives extra hardness (crushing strength) to the granules such that the crushing strength of -6+7 Tyler mesh, (3.4mm to 2.8mm in diameter) materials vary from 8 lbs of force to 10 lbs of force without the addition of urea formaldehyde as a hardening and conditioning agent. This is due to using concentrated urea of 95%, and spray agglomeration granulation. In comparison, typical commercial urea with 0.3% to 0.5% urea formaldehyde at -6+7 Tyler mesh (3.4mm to 2.8mm in diameter) has a hardness (crushing strength) of 5 to 8 lbs of force, but without formaldehyde, are much weaker.

Urea hardness (crushing strength) varies directly in a straight line manner with granule diameter, with a curve of the type $y=mx+c$, where y = the hardness, x = the diameter of the granule, m = the slope of the curve, and c = the intercept of the x -axis. Using this curve equation with the normal intercept as determined by classical data at 0.75, we can predict the hardness (crushing strength) of our urea/corn starch product to range 11 to 14 lb of force when the granules are 4mm in diameter and 0.9 to 1.1 lb of force when the granules are 1mm in diameter.

With regard to the use of urea formaldehyde as the
recognized manner of preventing urea caking during storage and
shipment, we have used some urea pretreated with 0.4% urea
formaldehyde in our tests to determine any positive or adverse
5 effect its presence might have on the controlled release
characteristics of our material. Some may wish to re-
granulate urea by melting or dissolving standard commercial
product or they may wish to add urea formaldehyde to resist
caking or other reasons. To demonstrate this was possible we
10 did some limited testing. In our test work, we were able to
make a product with some increased extension to the release
rate.

To measure the relative solubilities of the products in
soil, an irrigated soil burial test was devised such that
15 granules could be retrieved for measurement of their nitrogen
content. The following is a description of the test.

Procedure for Controlled Release Soil Test

1. Screen the sample to obtain -6+7 Tyler mesh granules
20 for the test.
2. Label a freezer container with the test description.
3. Place the freezer container on the 1200g balance and
tare out.
4. Place 300g of potting soil with a 40% moisture content
25 into the container and record the weight.

5. Over the soil place two (2) pieces of fiberglass mesh with 14 meshes to the inch and 1/16 inch openings.
6. Tare out the container with the soil and fiberglass mesh screens.
- 5 7. Spread 5 grams of -6+7 Tyler mesh granules over the screen in a single layer and record the weight.
8. Place a large square of fiberglass mesh over the granules, with a stainless steel screen cut to fit over it, so that the shape of the container has been mirrored.
- 10 9. Once this is shaped, tare out the container and add 150 g of soil and record the weight.
- 10 10. Repeat this process for each sample to be tested (in triplet if possible).
- 10 11. After all containers are completed, fill a mist spray bottle with de-ionized water and prime.
- 10 12. Tare out the weight of the primed mist bottle.
- 10 13. Mist 4g of water into each container and immediately place the lid on container and seal.
- 20 14. After the fertilizer granules have been submerged in a humid soil environment for the allotted time (9 hours, 24 hours, and 3 days), the 150g of soil is removed from the container.
15. Weigh to the nearest .0001g in aluminum weigh pan and tare out.
- 25 16. Gently remove the two pieces of fiberglass mesh, which contains the remaining fertilizer granules.

17. Transfer the granules to the aluminum weigh pan and record the weight of the fertilizer granules.
18. Place the fertilizer granules in a laboratory oven to dry at low temperature (50°C) for 13 hours.
- 5 19. Remove the dry sample from the oven, weigh to the nearest .0001g and record weight.
20. Place the dry fertilizer sample into a 125mL plastic sample bottle containing 20g of de-ionized water.
21. Allow the sample to dissolve for 3 hours.
- 10 22. Place approximately 1 ml aliquot of the sample solution onto the sample stage of a refractometer(e.g., Abbe Refractometer).
23. Record the refractive index and temperature of the solution.
- 15 24. Calculate the percent urea retained from the original fertilizer sample.

Using the above procedure, plain urea particles went into solution in the first 9 hours. Perlite granules containing urea and 1% corn starch and made from 85% urea melt retained up to 42% of their nutrient after 9 hours, 23% after 24 hours, and 11% after 3 days, thus providing an extended control release pattern. Further extended control release of the 25 granules resulted when 1% cornstarch was used as a gelling compound with a 95% urea melt; up to 48% of the nutrient

remained in the perlite after 9 hours, 23% remained after 24 hours, and 11% remained in the perlite after 3 days.

This is much less controlled retention than the goal of most sulfur coated ureas and methylene ureas, which are relatively expensive, longer nutrient availability extending materials.

Alternatively, cornstarch and cold water (33°F - 43°F) can be blended at ratios of as little as 1 to 1 (i.e. cornstarch is equal to or less than 50%) and then mixed with the urea melt before the absorption step of the process and thus avoid the homogenizer step in the process. This, however, adds water to the melt which must be dried out of the product, and for a continuous plant process would not be desirable.

While urea was employed in the tests as the principle source of nitrogen, diammonium phosphate (DAP) was additionally used as a source of nitrogen, as well as a source of phosphorus.

The control release fertilizer of the present invention was applied to outdoor plots of grass as described in Example 16. Two sample embodiments of the present controlled release fertilizer were prepared using urea, corn starch and expanded perlite. One sample fertilizer was prepared using a 1% corn starch solution and the second sample fertilizer was prepared using a 4% corn starch solution. An 85% urea solution was employed in preparing both the 1% and 4% sample fertilizers.

Test results show that the controlled release fertilizers provided the shortest time from planting to tasseling and silking for both sweet corn and field corn.

TABLE 1

CHEMICAL NAME

- 2-(2-Methyl-4-chlorophenoxy)propionic acid
- 2-Methyl-4-chlorophenoxyacetic acid
- 5 • 3,6-Dichloro-o-anisic acid
- Pyrethrins
- 2-chloro-4-ethylamino-s-triazine
- Benefin: N-butyl-N-ethyl-alpha, alpha, alpha, trifluoro-2, 6-dinitro-p-toluidine
- 10 • Trifluralin: alpha, alpha, alpha, trifluoro-2, trifluoro-2, 6-dinitro-N, N-dipropyl-p-toluidine
- Dithiopyr 3, 5-pyridinedicarbothiocic acid, 2-(difluoromethyl)-4-(2-methylpropyl)-6-(trifluoromethyl)-S, S-dimethyl ester
- 15 • Chlorpyrifos [O,O-diethyl-O-(3,5,6-trichloro-2-pyridyl)phosphorothioate]
- O,O-Diethyl S-(2-(ethylthio)ethyl)phosphorodithioate
- (2,2,2-trichloro-1-hydroxethyl)phosphonate
- 1-((6-chloro-3-pyridinyl)methyl)-N-nitro-2-
- 20 imidazolidinimine
- Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropane carboxylate
- (2,4,6,8-tetramethyl-1,3,5,7-tetraoxycyclo-octane)
- Prodiamine, (N3, N3-Di-n-propyl-2,4-nitro-
- 25 6(trifluoromethyl)-m-phenylenediamine)

More specifically, our invention encompasses taking urea melt of concentrations 40% to 99.9%, or more preferably 65% to 99.9%, and most preferably 75% to 99.9% made by any means and corn starch made by a means and blending them together into a 5 completely homogeneous mixture and in such a way that the gelling properties of the corn starch are not destroyed and foam formation is minimized. We blend under atmospheric pressure and do not let the temperature of the mixture exceed 295°F or a point where the vapor pressure of the mixture exceeds 450 mm of Hg while maintaining the temperature of the mixture above the point of first crystallization for urea. More preferably, we do not exceed 280°F or a point where the vapor pressure of the mixture exceeds 350 mm of Hg and most preferably we do not exceed 270°F or a point where the vapor pressure exceeds 300 mm of Hg. This prevents foaming which hinders the later absorption step, limits formation of biuret, 10 and limits thermal damage to the corn starch.

We minimize the mixing step and use only enough homogenization to completely mix the corn starch within the 20 urea solution. We use urea solution with more than 40% urea content up to 99.9% urea; however, to provide a more dense product and to get better extension of the release, we more prefer to use urea solution with a urea content between 65% and 99.9% and most prefer a urea solution between 75% urea and 25 99.5% urea. Further, we keep the melt at least 0.5°F above the point of first crystallization for the urea/corn starch

mixture; however, we prefer to keep it at least above 2°F, and
most prefer to keep it at least 5°F above the point of first
crystallization. Once the mixture has been made, it is
important to quickly absorb it to prevent damage to the corn
5 starch gel and to prevent excessive biuret formation. We pump
and meter the mixture without temperature adjustment into a
pugmill where it is mixed with the absorbing exfoliated
perlite. Although others who utilize our technology may wish
to adjust the temperature, we find temperatures adjusted at
10 this point can cause foaming or crystallization which at this
point are very harmful in obtaining maximum absorption into
the expanded perlite. Expanded perlite by any of most
standard means is heated to above the point of first
15 crystallization of the mixture to avoid premature freezing of
the mixture in the outer chambers of the perlite and thus
prevent full penetration. The metered perlite can be heated
by a fluid-bed or any number of ways and passed to the
absorber, however, we prefer to provide a secondary step of
20 limited exfoliation to the perlite as follows for much better
absorption and controlled release. A mixture of perlite and
water may be heated to steam the perlite, or hot steam may be
introduced directly to the perlite to steam the perlite.

The preferably hot steam filled perlite is fed to the
absorber where it absorbs the mixture to near completeness.
25 More urea/corn starch mixture is used than the absorbing
capacity of the perlite so that the perlite is essentially

totally submerged in the urea/corn starch mixture. This allows the excellent penetration and fill of the perlite particles. To give the mixture time to completely penetrate into the perlite before being crystallized or gel setting the
5 absorbers side walls are heated at the same temperature as the perlite-slurry and the top is covered to prevent evaporation. Although we do this in continuous fashion in a pugmill, it is obvious to those schooled in the art that some other absorber vessels may work just as well or to a limited degree as long as early crystallization of the mixture is not allowed. There must be excess in urea/corn starch over that which absorbs for this mixture is used as the mortar which covers and joins the individual pieces of perlite, now partially or totally filled with the urea/corn starch mixture, together into granules made up of a multiplicity of these filled perlite particles.
10 Retention time in the absorber can be from 10 seconds to several hours, however, we prefer to provide the time to obtain maximum penetration and yet minimize the time with respect to avoiding excess formation of biuret and damage to
15 the corn starch gel. Thus we more prefer 30 seconds to 30 minutes within the absorber, and most prefer 1 minute to 15 minutes within the absorber.

Once the urea/corn starch mixture is absorbed into the exfoliated perlite to the extend desired, the mixture is still
20 a slurry of urea/corn starch containing perlite in a mixture of urea and corn starch, as such it is pumped by mechanical,

pressure or suction means into the granulator. We have found that a coarse dispersion spray such as is used in most commercial drum granulators is preferred although we have been successful in pouring the material into the rolling bed of 5 granules and in pressure spraying the material with steam. When doing this, recycle as undersize and milled oversize and product, if needed, is fed back to the drum to provide cooling as needed and to assist in particle formation and agglomeration. Much of the cooling is provided by the 10 evaporation of water from the granules. We have found that the best temperature for granulation is to provide entering recycle at from 110°F to 220°F, but more preferably between 130°F and 210°F, and most preferably, between 150°F and 205°F, with the perlite/corn starch slurry fed into the drum at from 15 32°F to 295°F, but more preferably, from 115°F to 280°F, but most preferably, between 160°F and 270°F, but not allowing the temperature of the granules in the drum to exceed 235°F. The 20 rolling action and spraying action combine to form hard spherical granules with a good gel structure and with controlled release properties.

The difference between normally expanded perlite and exfoliated perlite as taught by our invention is shown by the following photo micrographs. Figure 2 shows the expanded perlite to be spherical to oblong in shape with an average 25 size of about 0.5 millimeters = 500 microns. Some openings are apparent on some of the material, but most of the

particles appear to be covered over with a thin shell. After steam exfoliation within the pugmill of the pilot plant, as shown in Figure 3, the outer covering is efficiently removed revealing the expanse of the internals. In essence, totally 5 changing the absorbency of the perlite. A close up of an exfoliated granule in Figure 4 shows just how open the perlite is to penetration by the urea and/or urea/corn starch mixture. A close up photo micrograph of expanded perlite, as in Figure 5, in contrast to Figure 4, shows limited exposure to the perlite internals to the urea or urea/corn starch mixtures. It also reveals a much easier means of egress by the absorbed mixture when it is in the soil, thus allowing the control and extension of nutrient release by the amount and type of blocking agent mixed with the urea or other nutrient.

With reference to Figure 1, one embodiment of the process of our invention includes taking fertilizer nutrient as a solution or as a melt and homogenously mixing it with a gelling material, i.e. blocking agent, in vessel (1) containing a high sheer homogenizer. However, if the mixture 20 is, e.g. a starch or similar material, and the solution is relatively cold, a homogenous mixture of the solution and the blocking agent can be obtained with less mixing force.

The homogenous solution is then pumped in a continuous manner by a metering pump (2) to a blender (3) to mix with an 25 absorbent. The absorbent is likewise continuously fed to the blender by being metered by a solids feeder (4) to a blending

type heat exchanger (5) to which water is also metered through a pump (6) and added to the absorbent prior to complete heating of the absorbent and in a manner that it is evenly dispersed among and within the particles of the absorbent.

- 5 Heat (7) is applied indirectly to the absorbent and water in the heat exchanger in a controlled manner to cause the water to expand to steam as the absorbent passes through the heat exchanger, this prepares the absorbent for maximum absorbency when it reaches the blender (3). Heat (8) is applied to the
- 10 blender to individually heat the contents and maintain good temperature control for optimum absorbency. In the blender the absorbent absorbs the mixture prepared in vessel (1) but not all of it; leaving an essentially filled absorbent with excess of that mixture in a very viscous but flowable condition to be discharged from blender (3) to feeder (9).
- 15 Thereby it can be introduced into the granulator (10) by a number of means. The filled absorbent particle with the absorbent mixture are granulated within the granulator such that the mixture crystallizes both within the absorbent
- 20 particles and outside the absorbent particles, the latter thus acting as the glue to hold the individual particles together into the form of a granule containing many particles. The granules discharge from the granulator after the particles and their contents and the accompanying mixture, making up the
- 25 granules, are solidified by the loss of heat and/or increase concentration. The heat of crystallization is removed by

- incoming recycle provided by the undersize from a sizing screen (11) and/or cooling gases passing through the granulator and/or heat losses passing through the shell of the granulator and/or by evaporation of water or other solvent
- 5 from the granules or evaporation cooling from other means within the granulator. In some cases heat will replace cooling to evaporate the solvent, thus increasing concentration of the mixture, both within and outside the absorbent, and resulting in solidification of the mixture.
- 10 Within the granulator, the particles from feeder (9), not only agglomerate among themselves, they also build on and agglomerate with the incoming recycle of undersize. Discharge from granulator (10) then free flows to screen (11) where the oversize is separated and sent to a mill (12) and then back to the screen (11). As an option to allow the best sphericity product, the milled material is all returned to the granulator. The on-size material leaving screen (11) free flows to a dryer/cooler (13) where it is dried to the desired completeness and cooled to a proper storage temperature.
- 15
- 20 Optionally, portions or all of the undersize and milled oversize can be returned to the blender (3) as is needed to improve granulation.

More specifically, we prefer that the heat exchanger (3) be a moderately high tip speed pugmill with heated sidewalls,

25 and that heat be provided by steam whose pressure at saturation can be easily regulated for a constant temperature

control. The heat exchanger (3) should be vented but only to let out the air and steam which would otherwise build to a pressure condition within the heat exchanger. We prefer to maintain as much as possible a steam atmosphere within the 5 pugmill, which is produced by evaporation of the water dispersed into the absorbent, and to discharge the exfoliated and/or steam containing absorbent directly to the blender (3). The blender is preferred to be a pugmill with moderate to slow tip speed, such that the mixing is gentle but thorough. The 10 material should reach a moderate oatmeal consistency as it exits the pugmill blender (3). We prefer the feeder (9) to be a low pressure developing pump or screw conveyor.

In other feeding means, we have been successful with a steam eductor whereby the filled absorbent and excess mixture 5 is sprayed onto the granules in the granulator. The granulation system which consists of the granulator, screen, mill and drying and cooling means and associated supporting equipment can be most any classical commercially existing system including spray drum granulators, pan granulators, 20 pugmill granulators, pour and crumble granulators, fluid-bed granulators, prill towers, and other forms of solid forming operations. The process is designed such that only minimal alterations are required to most every large (equal to or greater than 5 tons/hr) granulation plant now in operation 25 which produce granules or prills of urea, monoammonium phosphate, diammonium phosphate, sulfur, ammonium sulfate, and

ammonium nitrate, potassium nitrate, calcium nitrate, potassium phosphate, sodium nitrate, and mixtures of these products and others.

The following examples show how the present invention has
5 taken the above concepts and developed them into a unique extended release agricultural product and method of making and using same.

Thus, the invention is demonstrated with reference to the following examples, which are of an illustrative nature only
10 and which are to be construed as non-limiting.

EXAMPLE 1

Samples of controlled release urea were granulated using
15 an 85% urea solution, with and without corn starch equal to 1% of the final product, and pre-heated perlite 3-S. The urea and corn starch were combined in a laboratory beaker. A laboratory scale homogenizer was used to evenly disperse the corn starch in the urea solution. In separate tests, a
20 sufficient amount of perlite, both pre-heated to 300°F and unheated, was added to the urea/corn starch mixture to obtain almost complete absorption of the mixture. The mixture was removed from the beaker and allowed to solidify. Once the mixture had solidified and cooled, it was crumbled using a
25 laboratory blender on the chop setting, and then screened to obtain -6+7 Tyler mesh (3.4mm to 2.8mm in diameter) fertilizer

granules. These granules were then dried in a laboratory fluid-bed. The resulting materials were evaluated by placing 1 gm of sample in a test tube with 6 grams of water held at 75°F for 1, 2, and 3 days, at which time the samples were 5 drawn out of the test tube using a pipette after rotating the test tube end on end three times to create a homogenous solution. Urea retention in the perlite in all cases was over 250% better when it contained 1% corn starch instead of no corn starch and at least 35% better in all cases when the 10 perlite was heated.

EXAMPLE 2

A pilot plant was set-up where urea was melted by a steam tube melter then blended with water to make an 85% solution and continuously fed at 109 lb/hr to a mix tank equipped with a homogenizer where corn starch powder was added at the rate of 1 lb/hr. The urea solution and the mix tank were maintained at a temperature of 210°F. Expanded 3-S perlite 15 was continuously fed to a fluid-bed pre-heater at 7 lb/hr where it was heated with air until it was 320°F to 327°F. (No water was applied to the perlite before hand and no steam was used to exfoliate it.) The perlite and the urea/corn starch mixture were then fed to a pugmill where most of the urea/corn 20 starch mixture was absorbed while being held at a temperature of 196-197°F. The resulting slurry of perlite containing urea 25

and corn starch plus excess urea and corn starch mixture was fed to a second pugmill. Oversize granules produced during the pilot plant operation were milled utilizing a Jacobson knife-bladed hammermill to obtain additional product size
5 material and recycle. Recycle was added to the pugmill at a rate slightly over 2.5 to 1 that of the product made. The temperature of product leaving the pugmill was 136°F. The product and recycle were rounded and pre-dried in a rotating drum at 130°F after which the product was dried in a fluid-bed
10 dryer using 140°F air.

The resulting product had a bulk density of 26 lb/ft³, a perlite content of 8.8%, and a corn starch concentration of 1% giving a nitrogen content of 41.5+%; which resulted in a 9 hour dissolution rate in the aforementioned soil test of 43%,
15 23% after 24 hours, and 10% after 3 days.

EXAMPLE 3

Eighteen (18) grams of expanded 3-S perlite was placed in
20 a laboratory vessel having an agitator and small vent. 20 ml of water were added to the vessel and mixed with the perlite, and it was heated so that it steamed for 1 hour at 220°F. 350 grams of a mixture of 85% urea solution with 1% of corn starch homogenized with it was added to the steaming perlite and
25 mixed well. The mixture was poured onto a plastic surface to harden and then crumbled in a lab blender. The crumbled

material was screened to -6+10 Tyler mesh (3.4mm to 1.7mm in diameter) and dried in a lab fluid-bed. The resulting material had a bulk density of 35 lb/ft³. The material was then placed in a rotating drum and rounded by blowing hot air 5 on it at 240°F. The bulk density of the resulting material was 38 lb/ft³.

EXAMPLE 4

10 Eighteen (18) grams of expanded 3-S perlite was placed in a laboratory vessel and treated in the same manner as Example II except 350 grams of a 95% urea-1% corn starch mixture was added to the steaming perlite and mixed well. After 15 crumbling, screening, and drying, the resulting material had a bulk density of 35 lb/ft³ and after rounding, a bulk density of 37 lb/ft³.

EXAMPLE 5

20 The same test was performed as Example 3, but a 98% urea-1% corn starch mixture was added to the steaming perlite. The resulting material had a bulk density of 38 lb/ft³ and after rounding, a bulk density of 40 lb/ft³.

EXAMPLE 6

The same test was performed as Example 3, but a pure urea melt was added to the 18 grams of steam perlite resulting in a
5 bulk density of 41 lb/ft³ and after rounding 43 lb/ft³.

EXAMPLE 7

The apparatus of Example 2 was altered to allow
10 additional exfoliation of the expanded perlite in order to get
increased absorbency and increased bulk density per lab
examples 3, 4, 5, and 6. The perlite was fed into a double
shaft pugmill heated by a steam jacket at 85 psia or 316°F.
The shafts were rotated at 130 rpm to give them a tip speed of
15 3.4 ft/sec. As the perlite was metered to the pugmill, it was
moistened at the rate of approximately 1.1 grams of perlite
per gram of water at the inlet end of the pugmill to allow
absorption of the water into the perlite before the water was
heated to the point of becoming steam. The water was applied
20 through a tygon tube which dripped on the most active part of
the bed in the pugmill. Retention time of the perlite in the
pugmill was about 30 minutes. Photo micrographs showed the
perlite exiting the pugmill to have enhanced exfoliation of
the outer shell. The perlite was introduced to the urea/corn
25 starch mixture in a second pugmill with its double shaft
running at 72 rpm for a tip speed of 0.98 ft/sec. The

temperature of the perlite-urea/corn starch mixture was controlled by a steam jacket at 271°F through the use of 45 psia steam. The urea/corn starch mixture was prepared by melting granular urea and diluting it with water to 95% solution in the same mix tank as corn starch was homogenously blended into the mixture. The homogenizer operated at 3130 rpm and was powered by a 2 hp motor. The mixing was done in a semi-continuous manner. Residence time in the mixing tank was about 14 minutes during which it was under constant homogenization. Every 3 to 4 minutes, some of the mixture was withdrawn from the mixing vessel and put into a pump tank to provide continuous feed to the pugmill absorber. Once the withdrawal had occurred, additional amounts of urea and water to give a 95% urea solution were added to the mix vessel and corn starch was gradually poured into the vessel. The steam to the melter was 115 psia; however, temperatures of the mixing vessel was controlled at 269°F. In another change from Example 2, the perlite-urea/corn starch slurry leaving the absorber was sprayed by means of a steam eductor onto a rolling bed of granules in a rotating drum. The second pugmill mentioned in Example 2 was removed and the recycle and slurry were fed directly to the 4 ft dia. drum which was rotating at 15 rpm. Feed rate of urea @ 95% solution was 100.8 lb/hr with a corn starch feed rate of 1 lb/hr. Perlite fed at 4.2 lb/hr and recycle was fed back to the granulation drum at 27 lb/hr. Bed temperature within the granulation drum

was controlled at 217°F by means of blowing hot air at 227°F onto the rotating bed. Material discharged by the drum was fed to a vibrating type screener for separation into product, oversize, and undersize. The undersize and oversize milled by 5 a knife-bladed hammermill was fed back to the drum.

Granulation was excellent, forming spherical granules and very little oversize. The product size granules of -6+10 Tyler mesh (3.4mm to 1.7mm in diameter) size were dried and found to have a bulk density of up to 43 lb/ft³. In the soil burial tests previously mentioned, 33%, 16%, and 6% of the urea remained in the perlite after 9 hours, 24 hours, and 3 days, respectively. After drying the product, actual perlite content was 5.2% and corn starch was 1%. Nitrogen content of the product was 43+%. The hardness (crushing strength) of the urea by the recognized TVA crushing strength test as taught by 10 TVA Bulletin Y-147 was 9+ lbs of force for -6+7% Tyler mesh (3.4mm to 2.8mm in diameter) granules. Gel formation around and within the granules however, did not appear as good as the laboratory products when they were viewed as submerged in a 15 watch glass filled with water and with a surface stereo microscope. The individual perlite particles separated to a larger extent than normal while in water rather than being bound together by the gel.

EXAMPLE 8

Using the same equipment as in Example 7 but with alterations to the operating conditions, the good gelling properties reappeared in the final product. The same feed rates were maintained as in Example 7 and the same method of operation was used for enhanced exfoliation. However, the pugmill rpm was reduced to 97 rpm and thus the tip speed was reduced to 2.5 ft/sec. The temperature maintained in the urea melting and corn starch homogenization steps were reduced; mix tank retention time was reduced to 3½ minutes and homogenization was reduced from 14 minutes to 1 minute. Temperatures in the mix tank were reduced to 258°F and that in the pump tank to 262°F. The urea melt temperature fed to the mixing vessel was reduced to 283°F and the pugmill absorber temperature was reduced to 266°F. The temperature to the perlite steaming pugmill was reduced to 313°F. Steam pressure in the slurry venturi nozzle was operated at 30 psig. The resulting bulk density of the -6+10 Tyler mesh product was 39 lb/ft³. Urea remaining after 9 hours in the perlite after the soil burial tests was 44%, 10%, and 4.5% for 9 hours, 24 hours, and 3 days, respectively.

EXAMPLE 9

A 95% urea solution was homogenized to contain 1% corn starch and then for the most part absorbed by perlite equal to

4.5% of the final product in the same equipment as in Example
7. However, the granulation of the material was done by
spreading the molten slurry onto the bed of the rotating drum
by hand through use of a ice scoop of the open-top half-pipe
5 style. The scoop allowed the material to be distributed
across the rolling bed of the drum simulating a coarse spray
discharge longitudinally across the rolling bed and falling
curtains of particles as presently experienced in the large
drum of a urea granulation plant. Otherwise the manner of
10 operation was like that of Example 8. Urea melt at 100% and
about 283°F was fed to the mixing vessel. The mixture
temperature was varied from 268°F to 255°F during the 4 hour
operation as water and then corn starch was blended into it to
make the aforementioned mixture. Feed rates for the urea,
15 water, and corn starch were 111 lb/hr, 6 lb/hr, and 1 lb/hr
respectively. There was essentially no heel left in the mix
tank between blends. Once the blend was made, it was
immediately discharged to the pump tank, thus providing
continuous feed for the absorber. The urea/corn starch
20 mixture was fed to the absorber along with the perlite which
had been further exfoliated just prior to its introduction to
the absorber. About 1 ml of H₂O per gram of perlite was
introduced to the feed end of the pugmill and allowed to
absorb into the perlite. It expanded into steam in the steam
25 heated pugmill operating at 320°F, thus further exfoliating
the perlite. The absorber was run at from 268°F to 255°F as

the operation progressed. Slurry leaving the absorber was discharged to hand operated ice scoops. The granulation was done in the 4 ft diameter by 18" long drum rotating at 3.5 rpm, but increased to 7.5 rpm as the operation progressed.

5 Some of the absorber discharge was put into a aluminum sheet and allowed to solidify in a slab. The drum recycle was 33 lb/hr and the temperature of the bed was maintained at between 192°F to 201°F using the recycle and the hot air blower for control.

10 Material from the drum was screened to a product of -6+10 Tyler mesh and the oversize milled without drying and recycled to the screen. Undersize was fed to the drum as the recycle. As the temperature was varied in the homogenizer vessel from 268°F to 255°F, the mixture changed from clear to opaque and the gel strength in the final product as observed by the stereo microscope increased significantly, as did the soil burial test results, which went from a urea retention in

15 the perlite of 33% urea and 13% in 9 and 24 hours respectively, to a retention of 47% urea and 23% urea in the

20 perlite in 9 and 24 hours respectively, as the test progressed. The bulk density was acceptable for the entire run but decreased with an increase in gel strength from 38 lb/ft³ to 36.5 lb/ft³.

EXAMPLE 10

The pilot plant of Example 9 was operated in the same manner and rates as the best means of Example 9. However, 5 corn starch was applied at a strength of only 0.5% of the mixture. The resulting -6+10 Tyler mesh (3.4mm to 1.7mm in diameter) product had an increased bulk density of 39 lb/ft³ and soil burial result showed 45%, 16%, and 6% of the urea retained after 9 hours, 24 hours, and 3 days respectively.

10
EXAMPLE 11

The pilot plant of Example 9 was operated in the same manner and rates as the best means of Example 9 except there 15 was no addition of corn starch. Although the 95% solution of urea was absorbed by the perlite, it could not be granulated in the drum. The material was weak and turned to dust in the rotating drum. The perlite urea slurry was successfully 20 poured out on an aluminum sheet and solidified as a slab. The material which was poured and solidified was milled into granules, but it created large quantities of dust and would be unacceptable in a plant operation.

EXAMPLE 12

A 95% urea solution was homogenized with corn starch to give a 6% corn starch product in the same manner as Example 1,
5 but no perlite was added. The material was slowly poured on a bed of rotating granules in a pan granulator and granulated. The resulting product which contained no perlite was screened to -6+10 Tyler mesh (3.4mm to 1.7mm in diameter) product and had a bulk density of 32 lb/ft³. In the soil burial, it had a
10 urea retention of 27%, 15% and 6% in the perlite after 9 hours, 24 hours, and 3 days respectively.

EXAMPLE 13

The same test was done as above but had only 1% corn starch in the final product. The final product of -6+10 Tyler mesh (3.4mm to 1.7mm in diameter) granules had a bulk density of 40 lb/ft³. In the soil burial tests, urea retention in the perlite was 10%, 3% and 1% after 9 hours, 24 hours, and 3 days
20 respectively.

EXAMPLE 14

Using a standard pressure cooker, but without pressure development, expanded perlite was moistened with water at 20 ml of H₂O/18 grams of perlite in the laboratory and the vessel
25 was heated to exfoliate the perlite. Urea containing the

customary 0.3% to 0.5% formaldehyde used to condition it in most agricultural operations, was dissolved in H₂O to make a 95% solution. Corn starch was homogenized into the urea solution at 1% by weight. The urea/corn starch mixture was 5 poured into the perlite such that the perlite content was 5% of the dried product and allowed to absorb the urea formaldehyde/corn starch mixture. The resulting material was poured onto an aluminum sheet to cool. Then it was crumbled with a laboratory blender on the chop cycle, screened to -6+10 10 Tyler mesh and dried. The resulting material had a bulk density of 33 lb/ft³ and in the soil burial test retained 51% urea, 31% urea, and 15% urea in the perlite after 9 hours, 24 hours, and 3 days respectively.

5 EXAMPLE 15

In the same manner as Example 14, material was produced in the laboratory where by urea, diammonium phosphate and potassium chloride were dissolved in water to make an 85% 20 solution of the nutrients. The solution at 240°F was added to perlite to contain 8% of the perlite which had been further expanded in the manner of Example 14. The resulting product had a nutrient content of 29% nitrogen, 3% P₂O₅, and 4% K₂O and a bulk density of 41 lb/ft³. It showed excellent physical 25 properties.

EXAMPLE 16

Established grass plots of 5 ft by 15 ft were all equally clipped on August 15, 2000 to prepare for the application of 5 fertilizers. On August 16, 2000, the selected fertilizer blends were surface applied onto the individual plots. The fertilizer blends were as follows:

- Commerical-1 Fertilizer (Vigoro) 29-3-4: derived from polymer coated urea; polymer coated sulfur coated urea, urea, diammonium phosphate, muriate of potash, ferrous sulfate, and ferric oxide and containing 7.3% slowly available urea nitrogen from polymer coated urea and polymer coated sulfur.

- Urea based blend to make a 29-3-4 fertilizer containing:

Urea =	59.85%
Diammonium phosphate =	6.55%
Muriate of potash =	6.45%
Ferrous sulfate and ferric oxides =	2.00%
Clay =	4.2 %
Limestone =	<u>20.95%</u>
TOTAL =	100.00%

- Commercial-2 Fertilizer (Scotts Turf Builder) 29-3-4: derived from monoammonium phosphate, urea, methylene ureas, muriate of potash and containing 8.7% slowly available 25 methylene diurea and dimethylenetriurea nitrogen.

- Urea-perlite- at 0.92% corn starch based blend to make a 29-3-4 fertilizer which consisted of the base of controlled release fertilizer as formulated to be:

Urea:	92.44%
5 Corn starch:	.92%
Perlite 3-S:	<u>6.64%</u>
TOTAL:	100.00%

and which made up 64.75% of the blend

10 the remainder of the blend containing

diammonium phosphate	6.55%
muriate of potash	6.45%
ferrous sulfate and ferric oxides	2.00%
clay	4.20%
5 limestone	<u>16.05%</u>
TOTAL	100.00%

- Urea-perlite- at 3.60% corn starch based blend to make a 29-3-4 fertilizer which consisted of the base of controlled

20 release fertilizer as formulated to be:

Urea:	89.94%
Corn starch:	3.60%
Perlite 3-S:	<u>6.46%</u>
TOTAL:	100.00%

25

and which made up 66.45% of the blend

the remainder of the blend containing	
diammonium phosphate	6.55%
muriate of potash	6.45%
ferrous sulfate and ferric oxides	2.00%
5 clay	4.20%
limestone	<u>14.35%</u>
TOTAL	100.00%

0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
230
240
250
260
270
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500
510
520
530
540
550
560
570
580
590
600
610
620
630
640
650
660
670
680
690
700
710
720
730
740
750
760
770
780
790
800
810
820
830
840
850
860
870
880
890
900
910
920
930
940
950
960
970
980
990
1000
1010
1020
1030
1040
1050
1060
1070
1080
1090
1100
1110
1120
1130
1140
1150
1160
1170
1180
1190
1200
1210
1220
1230
1240
1250
1260
1270
1280
1290
1300
1310
1320
1330
1340
1350
1360
1370
1380
1390
1400
1410
1420
1430
1440
1450
1460
1470
1480
1490
1500
1510
1520
1530
1540
1550
1560
1570
1580
1590
1600
1610
1620
1630
1640
1650
1660
1670
1680
1690
1700
1710
1720
1730
1740
1750
1760
1770
1780
1790
1800
1810
1820
1830
1840
1850
1860
1870
1880
1890
1900
1910
1920
1930
1940
1950
1960
1970
1980
1990
2000
2010
2020
2030
2040
2050
2060
2070
2080
2090
2100
2110
2120
2130
2140
2150
2160
2170
2180
2190
2200
2210
2220
2230
2240
2250
2260
2270
2280
2290
2300
2310
2320
2330
2340
2350
2360
2370
2380
2390
2400
2410
2420
2430
2440
2450
2460
2470
2480
2490
2500
2510
2520
2530
2540
2550
2560
2570
2580
2590
2600
2610
2620
2630
2640
2650
2660
2670
2680
2690
2700
2710
2720
2730
2740
2750
2760
2770
2780
2790
2800
2810
2820
2830
2840
2850
2860
2870
2880
2890
2900
2910
2920
2930
2940
2950
2960
2970
2980
2990
3000
3010
3020
3030
3040
3050
3060
3070
3080
3090
3100
3110
3120
3130
3140
3150
3160
3170
3180
3190
3200
3210
3220
3230
3240
3250
3260
3270
3280
3290
3300
3310
3320
3330
3340
3350
3360
3370
3380
3390
3400
3410
3420
3430
3440
3450
3460
3470
3480
3490
3500
3510
3520
3530
3540
3550
3560
3570
3580
3590
3600
3610
3620
3630
3640
3650
3660
3670
3680
3690
3700
3710
3720
3730
3740
3750
3760
3770
3780
3790
3800
3810
3820
3830
3840
3850
3860
3870
3880
3890
3900
3910
3920
3930
3940
3950
3960
3970
3980
3990
4000
4010
4020
4030
4040
4050
4060
4070
4080
4090
4010
4020
4030
4040
4050
4060
4070
4080
4090
4100
4110
4120
4130
4140
4150
4160
4170
4180
4190
4200
4210
4220
4230
4240
4250
4260
4270
4280
4290
4210
4220
4230
4240
4250
4260
4270
4280
4290
4300
4310
4320
4330
4340
4350
4360
4370
4380
4390
4310
4320
4330
4340
4350
4360
4370
4380
4390
4400
4410
4420
4430
4440
4450
4460
4470
4480
4490
4410
4420
4430
4440
4450
4460
4470
4480
4490
4500
4510
4520
4530
4540
4550
4560
4570
4580
4590
4510
4520
4530
4540
4550
4560
4570
4580
4590
4600
4610
4620
4630
4640
4650
4660
4670
4680
4690
4610
4620
4630
4640
4650
4660
4670
4680
4690
4700
4710
4720
4730
4740
4750
4760
4770
4780
4790
4710
4720
4730
4740
4750
4760
4770
4780
4790
4800
4810
4820
4830
4840
4850
4860
4870
4880
4890
4810
4820
4830
4840
4850
4860
4870
4880
4890
4900
4910
4920
4930
4940
4950
4960
4970
4980
4990
4910
4920
4930
4940
4950
4960
4970
4980
4990
5000
5010
5020
5030
5040
5050
5060
5070
5080
5090
5010
5020
5030
5040
5050
5060
5070
5080
5090
5100
5110
5120
5130
5140
5150
5160
5170
5180
5190
5110
5120
5130
5140
5150
5160
5170
5180
5190
5200
5210
5220
5230
5240
5250
5260
5270
5280
5290
5210
5220
5230
5240
5250
5260
5270
5280
5290
5300
5310
5320
5330
5340
5350
5360
5370
5380
5390
5310
5320
5330
5340
5350
5360
5370
5380
5390
5400
5410
5420
5430
5440
5450
5460
5470
5480
5490
5410
5420
5430
5440
5450
5460
5470
5480
5490
5500
5510
5520
5530
5540
5550
5560
5570
5580
5590
5510
5520
5530
5540
5550
5560
5570
5580
5590
5600
5610
5620
5630
5640
5650
5660
5670
5680
5690
5610
5620
5630
5640
5650
5660
5670
5680
5690
5700
5710
5720
5730
5740
5750
5760
5770
5780
5790
5710
5720
5730
5740
5750
5760
5770
5780
5790
5800
5810
5820
5830
5840
5850
5860
5870
5880
5890
5810
5820
5830
5840
5850
5860
5870
5880
5890
5900
5910
5920
5930
5940
5950
5960
5970
5980
5990
5910
5920
5930
5940
5950
5960
5970
5980
5990
6000
6010
6020
6030
6040
6050
6060
6070
6080
6090
6010
6020
6030
6040
6050
6060
6070
6080
6090
6100
6110
6120
6130
6140
6150
6160
6170
6180
6190
6110
6120
6130
6140
6150
6160
6170
6180
6190
6200
6210
6220
6230
6240
6250
6260
6270
6280
6290
6210
6220
6230
6240
6250
6260
6270
6280
6290
6300
6310
6320
6330
6340
6350
6360
6370
6380
6390
6310
6320
6330
6340
6350
6360
6370
6380
6390
6400
6410
6420
6430
6440
6450
6460
6470
6480
6490
6410
6420
6430
6440
6450
6460
6470
6480
6490
6500
6510
6520
6530
6540
6550
6560
6570
6580
6590
6510
6520
6530
6540
6550
6560
6570
6580
6590
6600
6610
6620
6630
6640
6650
6660
6670
6680
6690
6610
6620
6630
6640
6650
6660
6670
6680
6690
6700
6710
6720
6730
6740
6750
6760
6770
6780
6790
6710
6720
6730
6740
6750
6760
6770
6780
6790
6800
6810
6820
6830
6840
6850
6860
6870
6880
6890
6810
6820
6830
6840
6850
6860
6870
6880
6890
6900
6910
6920
6930
6940
6950
6960
6970
6980
6990
6910
6920
6930
6940
6950
6960
6970
6980
6990
7000
7010
7020
7030
7040
7050
7060
7070
7080
7090
7010
7020
7030
7040
7050
7060
7070
7080
7090
7100
7110
7120
7130
7140
7150
7160
7170
7180
7190
7110
7120
7130
7140
7150
7160
7170
7180
7190
7200
7210
7220
7230
7240
7250
7260
7270
7280
7290
7210
7220
7230
7240
7250
7260
7270
7280
7290
7300
7310
7320
7330
7340
7350
7360
7370
7380
7390
7310
7320
7330
7340
7350
7360
7370
7380
7390
7400
7410
7420
7430
7440
7450
7460
7470
7480
7490
7410
7420
7430
7440
7450
7460
7470
7480
7490
7500
7510
7520
7530
7540
7550
7560
7570
7580
7590
7510
7520
7530
7540
7550
7560
7570
7580
7590
7600
7610
7620
7630
7640
7650
7660
7670
7680
7690
7610
7620
7630
7640
7650
7660
7670
7680
7690
7700
7710
7720
7730
7740
7750
7760
7770
7780
7790
7710
7720
7730
7740
7750
7760
7770
7780
7790
7800
7810
7820
7830
7840
7850
7860
7870
7880
7890
7810
7820
7830
7840
7850
7860
7870
7880
7890
7900
7910
7920
7930
7940
7950
7960
7970
7980
7990
7910
7920
7930
7940
7950
7960
7970
7980
7990
8000
8010
8020
8030
8040
8050
8060
8070
8080
8090
8010
8020
8030
8040
8050
8060
8070
8080
8090
8100
8110
8120
8130
8140
8150
8160
8170
8180
8190
8110
8120
8130
8140
8150
8160
8170
8180
8190
8200
8210
8220
8230
8240
8250
8260
8270
8280
8290
8210
8220
8230
8240
8250
8260
8270
8280
8290
8300
8310
8320
8330
8340
8350
8360
8370
8380
8390
8310
8320
8330
8340
8350
8360
8370
8380
8390
8400
8410
8420
8430
8440
8450
8460
8470
8480
8490
8410
8420
8430
8440
8450
8460
8470
8480
8490
8500
8510
8520
8530
8540
8550
8560
8570
8580
8590
8510
8520
8530
8540
8550
8560
8570
8580
8590
8600
8610
8620
8630
8640
8650
8660
8670
8680
8690
8610
8620
8630
8640
8650
8660
8670
8680
8690
8700
8710
8720
8730
8740
8750
8760
8770
8780
8790
8710
8720
8730
8740
8750
8760
8770
8780
8790
8800
8810
8820
8830
8840
8850
8860
8870
8880
8890
8810
8820
8830
8840
8850
8860
8870
8880
8890
8900
8910
8920
8930
8940
8950
8960
8970
8980
8990
8910
8920
8930
8940
8950
8960
8970
8980
8990
9000
9010
9020
9030
9040
9050
9060
9070
9080
9090
9010
9020
9030
9040
9050
9060
9070
9080
9090
9100
9110
9120
9130
9140
9150
9160
9170
9180
9190
9110
9120
9130
9140
9150
9160
9170
9180
9190
9200
9210
9220
9230
9240
9250
9260
9270
9280
9290
9210
9220
9230
9240
9250
9260
9270
9280
9290
9300
9310
9320
9330
9340
9350
9360
9370
9380
9390
9310
9320
9330
9340
9350
9360
9370
9380
9390
9400
9410
9420
9430
9440
9450
9460
9470
9480
9490
9410
9420
9430
9440
9450
9460
9470
9480
9490
9500
9510
9520
9530
9540
9550
9560
9570
9580
9590
9510
9520
9530
9540
9550
9560
9570
9580
9590
9600
9610
9620
9630
9640
9650
9660
9670
9680
9690
9610
9620
9630
9640
9650
9660
9670
9680
9690
9700
9710
9720
9730
9740
9750
9760
9770
9780
9790
9710
9720
9730
9740
9750
9760
9770
9780
9790
9800
9810
9820
9830
9840
9850
9860
9870
9880
9890
9810
9820
9830
9840
9850
9860
9870
9880
9890
9900
9910
9920
9930
9940
9950
9960
9970
9980
9990
9910
9920
9930
9940
9950
9960
9970
9980
9990
10000
10010
10020
10030
10040
10050
10060
10070
10080
10090
10010
10020
10030
10040
10050
10060
10070
10080
10090
10100
10110
10120
10130
10140
10150
10160
10170
10180
10190
10110
10120
10130
10140
10150
10160
10170
10180
10190
10200
10210
10220
10230
10240
10250
10260
10270
10280
10290
10210
10220
10230
10240
10250
10260
10270
10280
10290
10300
10310
10320
10330
10340
10350
10360
10370
10380
10390
10310
10320
10330
10340
10350
10360
10370
10380
10390
10400
10410
10420
10430
10440
10450
10460
10470
10480
10490
10410
10420
10430
10440
10450
10460
10470
10480
10490
10500
10510
10520
10530
10540
10550
10560
10570
10580
10590
10510
10520
10530
10540
10550
10560
10570
10580
10590
10600
10610
10620
10630
10640
10650
10660
10670
10680
10690
10610
10620
10630
10640
10650
10660
10670
10680
10690
10700
10710
10720
10730
10740
10750
10760
10770
10780
10790
10710
10720
10730
10740
10750
10760
10770
10780
10790
10800
10810
10820
10830
10840
10850
10860
10870
10880
10890
10810
10820
10830
10840
10850
10860
10870
10880
10890
10900
10910
10920
10930
10940
10950
10960
10970
10980
10990
10910
10920
10930
10940
10950
10960
10970
10980
10990
11000
11010
11020
11030
11040
11050
11060
11070
11080
11090
11010
11020
11030
11040
11050
11060
11070
11080
11090
11100
11110
11120
11130
11140
11150
11160
11170
11180
11190
11110
11120
11130
11140
11150
11160
11170
11180
11190
11200
11210
11220
11230
11240
11250
11260
11270
11280
11290
11210
11220
11230
11240
11250
11260
11270
11280
11290
11300
11310
11320
11330
11340
11350
11360
11370
11380
11390
11310
11320
11330
11340
11350
11360
11370
11380
11390
11400
11410
11420
11430
11440
114

atmosphere temperatures and humidity, the plots were irrigated as required, approximately three times weekly.

Visual observation and harvesting of the grass were two methods used to evaluate the performance of the controlled 5 release fertilizer.

A greening rating of each plot was taken each Wednesday prior to cutting the grass and irrigating. The greening rating was based on a scale of 1 to 5 with 5 being the best possible and 1 the lowest rating. At the same time, the grass plots were examined for any indication of blade damage due to 10 too much fertilizer availability.

The grass clippings were weighed after each cutting. One grass sample from each type of fertilized plot was analyzed for the nitrogen content each week.

The grass greening test data is shown by plots and 5 fertilizer types and rates in Table 2. The first greening rating was made on August 23, 2000 exactly one week after the fertilizer application was made on August 16, 2000. The urea-perlite-0.92% corn starch and urea-perlite-3.60% corn starch 10 based fertilizers produced a quick greening of the grass and continued to perform in an excellent manner until the killing frosts of October 8th and 9th. In particular, the fertilizers containing the urea-perlite-0.92% corn starch based blend 15 maintained an excellent rating and at the conclusion of the trial on October 11, 2000, had an average rating of 3.27 on the A-4 plot and a 3.67 average rating on the B-6 plot. This 20 25

was overall superior to any other tested fertilizer when applied at the rate of 1 lb for nitrogen per 1,000 ft² of surface. There was never any evidence of blade damage due to excessive availability of the fertilizer.

TABLE 2
GRASS PLOTS
GREENING TEST DATA^{1,2,3,4}

Plot #	Plot Description	08/23/00	08/30/00	09/06/00	09/13/00	09/20/00	09/27/00	10/04/00	10/11/00	SUM	Avg
A-1	Commercial-1 Fertilizer 29-3-4 (1X)	2.68	2.33	3.00	3.55	3.10	2.33	2.65	1.50	21.14	2.64
A-2	Urea Blend (29-3-4) (1X)	2.93	2.50	2.93	3.68	3.00	2.35	2.65	1.50	21.54	2.69
A-3	Commercial-2 Fertilizer 29-3-4 (1X)	2.73	2.45	2.80	3.68	3.05	2.25	2.30	1.50	20.76	2.60
A-4	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (1X)	3.23	3.95	4.08	4.23	3.65	2.85	2.65	1.50	26.14	3.27
A-5	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (4X)	4.45	4.43	4.55	4.68	4.13	3.32	3.20	1.50	30.26	3.78
A-6	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (3X)	3.50	4.20	4.65	4.68	4.25	3.45	3.30	1.50	29.53	3.69
A-7	No Fertilizer	2.45	2.25	2.28	3.00	2.93	3.60	3.60	1.50	21.61	2.70
B-1	Urea-Perlite, at 3.60% Corn Starch (29-3-4) (1X)	3.25	2.70	2.88	3.75	3.23	2.88	3.70	1.50	23.89	2.99
B-2	Urea-Perlite, at 3.60% Corn Starch (29-3-4) (4X)	4.23	4.55	4.43	4.63	4.03	3.43	3.90	1.50	30.70	3.84
B-3	Commercial-1 Fertilizer 29-3-4 (1X)	2.95	2.55	2.88	4.05	3.03	3.00	3.15	1.50	23.11	2.89
B-4	Urea Blend (29-3-4) (1X)	3.00	4.03	3.78	4.05	3.65	3.18	3.25	1.50	26.44	3.31
B-5	Commercial-2 Fertilizer 29-3-4 (1X)	2.88	4.18	4.00	4.05	3.58	3.23	3.13	1.50	26.55	3.32
B-6	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (1X)	3.75	4.33	4.30	4.38	3.83	3.50	3.80	1.50	29.39	3.67
B-7	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (4X)	4.48	4.75	4.93	4.85	4.15	4.00	4.30	1.50	32.96	4.12
C-1	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (3X)	4.00	4.20	4.33	4.55	4.00	3.52	4.00	1.50	30.10	3.76
C-2	Urea-Perlite, at 3.60% Corn Starch (29-3-4) (1X)	3.63	3.98	4.13	4.35	3.35	2.95	3.90	1.50	27.79	3.47
C-3	Urea-Perlite, at 3.60% Corn Starch (29-3-4) (4X)	4.25	4.60	4.80	4.75	4.13	3.48	3.90	1.50	31.41	3.93

Note 1: A (1X) application rate is 1 pound of nitrogen per thousand square feet, 3X and 4X are 3 and 4 pounds of nitrogen per 1000 square feet respectively

Note 2: The ratings were made on seven day intervals

Note 3: Visual observation are on a scale of 1 to 5 (5 being the highest rating)

Note 4: Fertilizer application was applied on August 16, 2000

The cumulative wet weight grass clipping weights for the replicated plots are shown in Table 3. The urea-perlite and 0.92% corn starch based blend at 1X application rate, at the 5 conclusion of the trials on 10/11/00, had 97% more total weight produced than that produced by the urea based blend, 108% more than that produced by the Commerical-1 fertilizer 29-3-4 blend, and 48% more than that produced by the Commerical-2 fertilizer 29-3-4 blend. In addition, the urea-perlite-0.92% corn starch based blend maintained superior 0 grass growth over the entire duration of the eight week test.

In Table 3, the first number is the combined weights and the second number is the cumulative weights. The no fertilizer plot did not have a replicated plot.

TABLE 3^{1,2,3,4,5,6,7,8}

GRASS CLIPPING DATA(GRAMS)

PLOT DESCRIPTION ^{9,10}	8/23/00	8/30/00	9/6/00	9/13/00	9/20/00	9/27/00	10/4/00	10/11/00
ZERO FERTILIZER	15	54/69	38/107	66/173	53/226	54/280	18/298	6/304
UREA (29-3-4) (1X)	82	192/274	215/489	270/759	102/861	101/962	53/1015	13/1028
COMMERCIAL-1 FERTILIZER (29-3-4) (1X)	65	150/215	198/413	266/629	147/776	142/918	48/966	6/972
COMMERCIAL-2 LAWN FERTILIZER 29-3-4 (1X)	123	260/383	324/707	338/1045	162/1207	101/1308	60/1368	5/1373
UREA-PERLITE, AT 0.92% CORN STARCH (29-3-4) (1X)	228	481/709	497/1206	428/1634	145/1779	162/1941	75/2016	10/2026
UREA-PERLITE, AT 0.92% CORN STARCH (29-3-4) (4X)	487	1790/2277	1406/3683	1026/4709	465/5174	350/5524	79/5603	33/5636
UREA-PERLITE, AT 0.92% CORN STARCH (29-3-4) (3X)	66	600/666	1094/1760	919/2679	469/3148	264/3412	111/3523	23/3546
UREA-PERLITE, AT 3.60% CORN STARCH (29-3-4) (1X)	146	352/498	445/943	450/1393	142/1535	125/1660	81/1741	5/1746
UREA-PERLITE, AT 3.60% CORN STARCH (29-3-4) (4X)	449	1730/2179	1312/3491	1164/4655	322/4977	272/5249	167/5416	17/5433
SOIL TEMPERATURE ^{4,5}	72	69	75	65	66	61	57	52
SOIL MOISTURE ⁷	7.4	7.0	7.0	7.4	8.8	10.0	9.0	9.0

NOTES:

1. AVERAGE CLIPPING WEIGHTS FOR THE DUPLICATE PLOTS. THE CONTROL PLOT WAS NOT DUPLICATED.
2. TEST PLOTS WERE FERTILIZED ON AUGUST 16, 2000.
3. LENGTH OF CUTTING INTERVAL = SEVEN DAYS
4. HEIGHT OF CUTTING = 3 INCHES
5. SOIL TEMPERATURE IN DEGREE FAHRENHEIT (AVERAGE OVER SEVEN DAY PERIOD)
6. SOIL TEMPERATURE HAS A SIGNIFICANT CORRELATION ON 419 TIFTON BERMUDA GRASS GROWTH.
7. CUMMULATIVE WEIGHT FOLLOWS WEIGHT OF CLIPPINGS FOR EACH DATE.
8. SOIL MOISTURE WAS MAINTAINED BETWEEN 7 AND 10 UTILIZING A SOIL MOISTURE METER.
9. REFERS TO BASE MATERIAL USED IN THE N-P-K BLEND WHICH WAS APPLIED TO THE DUPLICATE PLOTS
10. ALL FERTILIZERS WERE BLENDED TO GIVE A 29-3-4 N-P-K ANALYSIS

The nitrogen concentration data by sample is shown in Table 4. Besides showing excellent total grass production on each cutting, the urea-perlite-0.92% corn starch blend at the base application rate of 1 lb per 1,000 ft² maintained excellent 5 nitrogen content. The test results clearly indicate that the urea-perlite-0.92% and 3.60% corn starch blends provide the ability not only to quickly green and then maintain grass green while preventing blade burn damage, but they also allow tremendous increase in growth and nitrogen recovery by this 0 grass and most likely many other grasses as well as other food and foliage producing vegetation.

TABLE 4
NITROGEN CONCENTRATION DATA BY PLOTS 1,2,3,4,5

Plot #	Plot Description	8/23/00	8/30/00	9/6/00	9/13/00	9/20/00	9/27/00	10/4/00
A-7	No Fertilizer	2.29%	2.15%	2.79%	2.81%	2.70%	2.76%	2.41%
B-3	Commercial-1 Fertilizer 29-3-4 (1X)	2.41%	2.83%	2.79%	2.70%	2.88%	2.68%	2.39%
B-4	Urea Blend (29-3-4) (1X)	2.50%	3.02%	3.25%	2.87%	3.05%	2.67%	2.48%
B-5	Commercial-2 Fertilizer 29-3-4 (1X)	2.73%	3.19%	3.05%	2.82%	3.00%	2.73%	2.47%
B-6	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (1X)	3.46%	3.57%	3.22%	3.20%	3.02%	3.13%	2.85%
B-7	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (4X)	4.20%	4.38%	4.19%	3.90%	3.24%	3.91%	3.66%
C-1	Urea-Perlite, at 0.92% Corn Starch (29-3-4) (3X)	3.35%	4.34%	3.70%	3.60%	3.53%	3.39%	3.12%
C-2	Urea-Perlite, at 3.60% Corn Starch (29-3-4) (1X)	3.10%	3.31%	3.07%	3.02%	3.11%	2.87%	2.67%
C-3	Urea-Perlite, at 3.60% Corn Starch (29-3-4) (4X)	3.85%	4.18%	4.09%	3.75%	3.93%	3.50%	3.34%

Note 1: Fertilizer application was applied on August 16, 2000

Note 2: 1X application rate represents 1 pound of nitrogen per thousand square feet

Note 3: Grass clippings from this duplicate test plot areas were analyzed for nitrogen content.

Note 4: Grass samples analyzed by Thornton Laboratories Tampa, Florida

Note 5: Grass clippings harvested on seven day intervals

EXAMPLE 17

In the same manner as Example 14, material was produced in the laboratory where by using a pressure cooker, but 5 without pressure development, expanded perlite was moistened with water at 20 ml of H₂O per 18 grams of perlite in the laboratory and vessel was heated to exfoliate the perlite. Urea was dissolved in H₂O to make a 95% solution. Unmodified wheat starch was homogenized into the urea solution at 1% by weight. The urea/wheat starch mixture was poured into the exfoliated perlite such that the perlite content was 5.2% of the dried product and allowed to absorb the urea/wheat starch mixture. The resulting material was poured onto an aluminum sheet to cool. Then it was crumbled with a laboratory blender on the chop cycle, screened to -6+10 Tyler mesh and dried. 15 The resulting material had a bulk density of 33 lb/ft³ and in soil burial tests, retained 61% of the urea after 9 hours and 30% after 24 hours.

20

EXAMPLE 18

Urea was dissolved in H₂O to make a 95% solution. Corn starch was homogenized into the urea solution at 1% by weight. The urea/corn starch mixture was poured into a vessel 25 containing newspaper which had been chopped in a laboratory blender to a near lint condition. The newspaper was not

steamed, wetted, or pre-heated before being exposed to the urea/corn starch mixture. The newspaper content was 3.5% of the final product. The resulting material was poured on an aluminum sheet to cool. Then it was crumbled with a 5 laboratory blender on the chop cycle, screened to -6+10 Tyler mesh and dried. The resulting material had a bulk density of 30 lb/ft³ and in a soil burial test, retained 46% of the urea after 9 hours and 17% of the urea after 24 hours.

EXAMPLE 19

Using a standard pressure cooker but without pressure development, expanded perlite was moistened with water at 20 ml of H₂O per 36 grams of perlite in the laboratory and the 15 vessel was heated to exfoliate the perlite. 700 grams of urea was dissolved in H₂O along with 3 grams of magnesium oxide such that the solution became 85% urea. The solution was poured into the vessel containing the 36 gram of exfoliated perlite and mixed. The resulting material was poured onto an aluminum 20 foil to harden. The hardened mixture was crumbled in the laboratory blender and screened to -6+10 Tyler mesh. The granules were dried. The bulk density was 28 lbs/ft³.

EXAMPLE 20

A urea/corn starch homogenous mixture was prepared in the laboratory using a 95% solution of urea and homogenizing corn
5 starch into the urea solution at 265°F to make a mixture containing 8% corn starch. The mixture was poured onto a metal pan and allowed to solidify after which it was crumbled using a laboratory blender on the chop cycle, screened, and dried. In the soil burial test, 62% of the urea remained in
10 the corn starch gel after 9 hours. The products bulk density was 41 lb/ft³.

EXAMPLE 21

In the same manner as Example 20, a mixture containing 6% corn starch was made and granulated by pouring it in a laboratory pan granulator. The resulting product was screened, dried, and soil tested. In the soil burial test, 25%, 21% and 15% of the original urea remained in the corn
20 starch gel after 9 hours, 24 hours, and 3 days, respectively. The products bulk density was 32 lb/ft³.

While only a few exemplary embodiments of this invention have been described in detail, those skilled in the art will recognize that there are many possible variations and
25 modifications which may be made in the exemplary embodiments while yet retaining many of the novel and advantageous

features of this invention. Accordingly, it is intended that the following claims cover all such modifications and variations.